The Superconductivity, Intragrain Penetration Depth and Meissner Effect of $RuSr_2(Gd,Ce)_2Cu_2O_{10+\delta}$

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The hole concentration p (δ), the transition temperature T_c, the intragrain penetration depth λ, and the Meissner effect were measured for annealed $RuSr_2(Gd,Ce)_2Cu_2O_{10+\delta}$ samples. The intragrain superconducting transition temperature T_c varied from 17 to 40 K while the p changed by only 0.03 holes/CuO_2. The intragrain superfluid-density $1/\lambda^2$ and the diamagnetic drop of the field-cooled magnetization across T_c (the Meissner effect), however, increased more than 10 times. All of these findings are in disagreement with both the T_c vs. p and the T_c vs. $1/\lambda^2$ correlations proposed for homogeneous cuprates, but are in line with a possible phase-separation and the granularity associated with it.

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One key question about $RuSr_2GdCu_2O_{8+\delta}$ (Ru1212) and $RuSr_2(Gd,Ce)_2Cu_2O_{10+\delta}$ (Ru1222), where the partially ferromagnetically (FM) aligned Ru-spins and the superconducting (SC) CuO_2 layers are structurally adjacent, is what determines their superconductivity. Different groups have emphasized either their underdoped nature (*i.e.* the hole-concentration $p << 0.16$ holes/CuO_2) or the competition between the SC and the coexisting FM through the Fulde-Ferrell-Larkin-Ovchinnikov (FFLO) phase.[1,2,3,4,5] A verification was difficult until the recent reports that the transition temperature T_c's of Ru1222 and Ru1212 can be adjusted over a broad range by oxygen-annealing and Cu-substitution, respectively.[6,7,8] The reported data, unfortunately, show that the T_c-enhancement is accompanied by both a p-increase and a suppression of the ferromagnetic spin-order, and offer no clear distinction. To explore the topic, we measured the intragrain T_c, the p, the intragrain superfluid-density $1/\lambda^2$, and the Meissner effect in several annealed Ru1222 samples. The data were then compared with both the T_c vs. p correlation,[9] and the T_c vs. $1/\lambda^2$ line proposed.[10] We observed that a two-fold enhancement of T_c (from 17 to 40 K) is accompanied by a relatively small change of p (from 0.09 to 0.12 holes/CuO_2), but a 20-fold increase in $1/\lambda^2$ (from 0.3 to 6 μm^{-2}). Together with an extremely large field effect of $dT_c/dH > 100$ K/T and a linear increase of the Meissner fraction with $1/\lambda^2$, the data suggest that those ruthenocuprate grains are actually Josephson-junction-arrays (JJA), in agreement with the phase-separation model suggested.[11] Further investigations of local magnetic structures are needed to solve the problem.

Ceramic $RuSr_2(Gd_{0.7}Ce_{0.3})_2Cu_2O_{10+\delta}$ samples were synthesized following the standard solid-state-reaction procedure. Precursors were first prepared by calcined commercial oxides at 400-900 °C under flowing O_2. Mixed powder with a proper cation ratio was then pressed into pellets and sintered at 900 °C in air for 24 hr. The final heat treatment of the ceramics was done at 1090 °C for 60 hr after repeatedly sintering and regrinding at 1000 °C. Powders with different particle sizes were prepared according to the procedures previously reported.[12] The structure of the samples was determined by powder X-ray diffraction (XRD), using a Rigaku DMAX-IIIB diffractometer. The XRD pattern of a typical sample is shown in Fig. 1. Refinement was done based on a space group of I4/mmm with lattice parameters of $a = 3.839(1)$ and $c = 28.591(5)$ using the Rietan-2000 program.[13] There are no noticeable impurity lines in the X-ray diffraction pattern within our resolution of a few percent. The grain sizes ($\approx$ 2-20 μm) of the ceramic samples, as well as the particle sizes of the powders, were measured using a JEOL JSM 6400 scanning electron microscope (SEM). The magnetizations were measured using a Quantum Design SQUID magnetometer with an *ac* attachment.

The data from nine Ru1222 samples in three sets are presented here. A0, B0, and C0 are as-synthesized samples with slightly different superconducting transition temperatures between 26 and 30 K (probably due to the slight differences in the final heat treatment and the various resulting intragrain granularities). All others are pieces of the respective as-synthesized ceramic after a 2 hr/600 °C anneal. The gases used in annealing are 300 atm. O_2 for samples A1, B1, and C1; 20 atm. O_2 for sample A2; Ar+0.01 atm. O_2 for sample C2; and high purity Ar (99.99%) for sample C3. The zero-field-cooled magnetization (M_{ZFC}) and the field-cooled one (M_{FC}) of samples A0, A1, and A2 are shown in Fig. 2. A systematic increase of the T_c (defined as the major inflection point of the M_{ZFC} and marked by arrows in the figure) with the assumed oxygen-intake can be clearly seen, *i.e.* from 26 to 40 K.[7] It should be pointed out that the T_c so-defined is actually the intragrain transition-temperature based on both the size- and the H_{ac}-dependencies of the *ac* χ measured in the same samples, where H_{ac} is the *ac* field used (Fig. 3).[12,14] The intergrain transition of our Ru1222 samples is typically $\approx$ 10-20 K lower, and a prefect shielding can be reached only under $H_{ac} \approx 0.01$ Oe (Fig. 3). This relatively weaker intergrain coupling of Ru1222 ceramic seems to be typical in

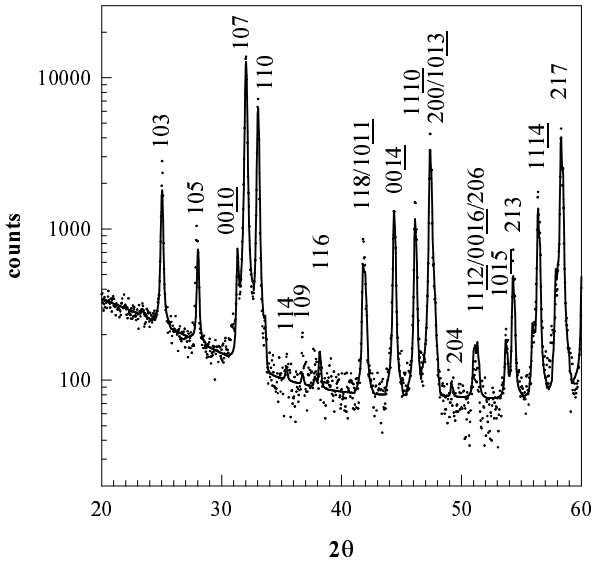

FIG. 1: The XRD of sample A0. dots: data; solid line: the refinement with I4/mmm.

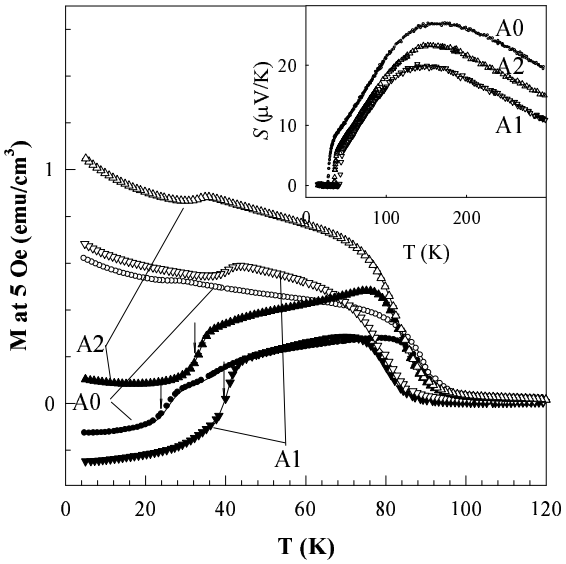

FIG. 2: The M_{ZFC} (solid symbols) and M_{FC} (open symbols) at 5 Oe for ◯/●: sample A0; ▽/▼: sample A1; and △/▲: sample A2. Inset: Thermal power S(T) of the three samples.

previously published reports, where a less than 100% shielding in M_{ZFC} was observed.[7,15] We attribute this to the 1090 °C final heat-treatment temperature used, which is higher than that used for Ru1212.

To estimate the p, the thermoelectric power, S, was measured (inset, Fig. 2). The overall T-dependence of the S is similar to that of the underdoped $YBa_2Ca_3O_{7-\delta}$, and is in agreement with the data previously reported.[15] No evidence of the RuO_2 contributions can be noticed.[1] A moderate increase of p (*i.e.* from 0.104 holes/CuO_2 in sample A0 to 0.121 holes/CuO_2 in sample A1) was then deduced using the proposed universal correlation of S(290 K) = 992exp(-38.1p).[16] To verify the deduced p,[15] the oxygen-intake, $\Delta\delta$, was measured using a gas-effusion cell, where the sample was heated to 800 °C and the released oxygen was measured by both a pressure-gauge and a mass spectrometer.[17] The total oxygen released is 0.1 and 0.115 O/Ru1222 for samples A0 and A1, respectively. While the absolute stoichiometry, 10+δ, may depend on the phase compositions at 800 °C, the $\Delta\delta$ should be less debatable. The

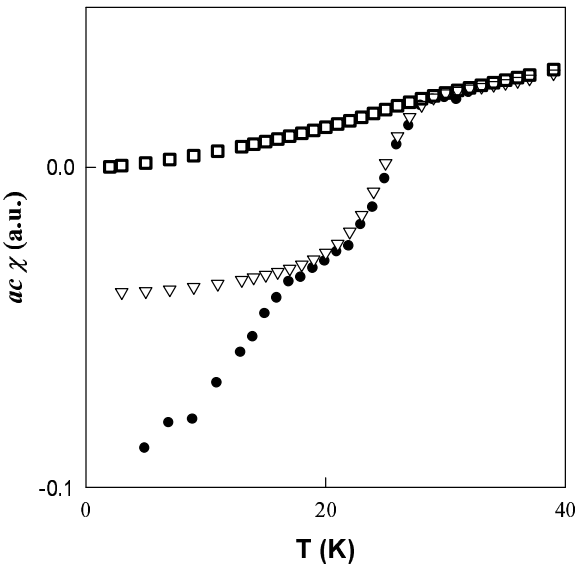

FIG. 3: $ac\chi$ of the bulk sample A0 (powders with particle size of 10 μm or larger show the same χ) under H_{ac} of 3 ($\bigtriangledown$) and 0.01 Oe ($\bullet$), as well as that at H_{ac} = 3 Oe of a 1 μm powder made from it ($\square$).

$\Delta\delta \approx 0.015$ so obtained is in good agreement with both the estimated p from S and the reported rate of $\Delta\delta/\Delta T_c \approx 0.0014$ O/K for Ru1222 with similar T_c.[18,19] The expected T_c enhancement, however, should be less than 6 K based on the correlation of $T_c = T_{c,\max}[1\text{-}82(p\text{-}0.16)^2]$ with $T_{c,\max} \leq 50$ K.[16] This value is far smaller than the 14 K enhancement observed, and the change in carrier concentration should not be the dominant factor.

It is interesting to note that the T_c-enhancement is accompanied by an increase in the diamagnetic drops (ΔM_{ZFC}, ΔM_{FC}) across T_c in both M_{ZFC} and M_{FC} . For example, the ΔM_{ZFC} is $\approx$ 0.15, 0.2, and 0.3 emu/cm^3 at 5 Oe for samples A0, A2, and A1, respectively (Fig. 2).[20] Comparable trends can also be noticed in the data previous reported.[7] We tentatively attribute the change in ΔM_{ZFC} to a decrease in $1/\lambda^2$ (actually $1/\lambda_{ab}^2$), and verify the interpretation by the direct measurement of the intragrain penetration-depths λ through the ac susceptibility of powders. Several powders were prepared from the same ceramic by sorted according to their particle sizes, and the λ was deduced from the size dependency of the χ observed. The details have been reported before.[12,21] The previous data analysis procedure, however, was slightly modified here to simultaneously fit both the large magnetic background χ_m and λ. For a randomly-oriented power j (=1...n), which contains particles $i = 1...m$ with sizes of $d_{j,i}$, one has:

$\chi_j = \sum_i \int\{[1 - 6(\lambda_{ab}/d_{j,i})coth(d_{j,i}/2\lambda_{ab}) + 12(\lambda_{ab}/d_{j,i})^2]d_{j,i}^3 cos^2\theta sin\theta$

$+[1-6(\lambda_c/d_{j,i})coth(d_{j,i}/2\lambda_c)+12(\lambda_c/d_{j,i})^2]d_{j,i}^3 sin^2\theta sin\theta\}d\theta/\sum_i d_{j,i}^3+\chi_m$, where λ_c, λ_{ab}, and θ are the penetration depths along c, ab, and the polar angle, respectively. When $\lambda_c >> \lambda_{ab}$ (highly anisotropy approximation) one has:

$\chi_j = \sum_i\{[1 - 6(\lambda_{ab}/d_{j,i})coth(d_{j,i}/2\lambda_{ab}) + 12(\lambda_{ab}/d_{j,i})^2]d_{j,i}^3/3\sum_i d_{j,i}^3 + \chi_m$.

A regression was used to calculate λ without assumptions of χ_m. The χ_j of the powder with the smallest average particle size was used as the initial value of χ_m. The initial value of λ was deduced from the χ of the powder with the largest particle size. The new χ_m was then regressively calculated through a least-square fit using the approximate λ value and the χ_j, $d_{j,i}$ data observed. The regression of λ followed. Typically, the result will be convergent to within 1% after three regression cycles.

The procedure was tested on the data of $YBa_2Cu_3O_{6.6}$ and MgB_2 previously collected, and led to a good agreement with the values expected.[12,21] The possible uncertainty of the λ seems to be less than 30%, mainly from the statistical uncertainty of d_i. As an example, the data of three powder-samples from ceramic A1 with effective particle-sizes of d = 1.9, 1.3, and 0.9 μm are shown in the inset of Fig. 4. Their χ, the deduced χ_m, and the fitting results are shown as symbols, a dashed line, and solid lines, respectively. The deduced superfluid densities, $1/\lambda^2$'s of samples A0 and A1 are shown in Fig. 4. The twofold increase of $1/\lambda^2$, which is in agreement with the raw M_{ZFC} data of both that in the Fig. 2 and that reported previously,[7,8] confirms the above assumption that the change in $1/\lambda^2$ is the dominant factor for ΔM_{ZFC} and ΔM_{FC}. This view is further supported by an unusual large $dT_c/dH \approx 100$ K/T similar to that observed in Ru1212Eu (Fig. 5).

The increase in $1/\lambda^2$ is significantly higher than that expected from both the p and the T_c observed. In principle, the effective mass m^*, pair-breaking scatterings, and intragrain granularity can all affect the $1/\lambda^2$ observed. However,

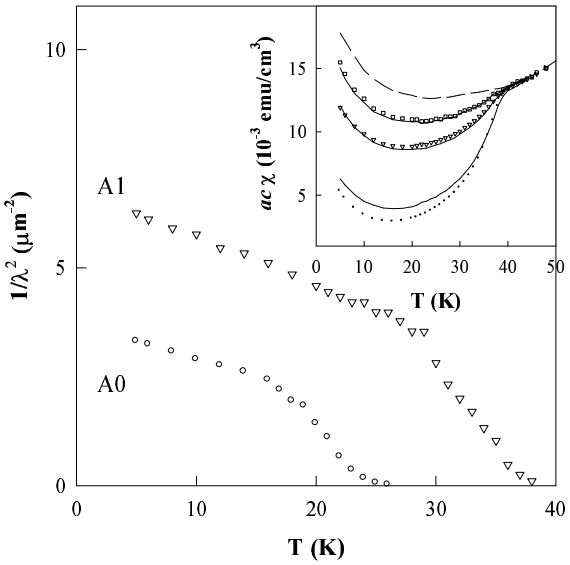

FIG. 4: The intragrain $1/\lambda^2$ for $\bigtriangledown$: sample A0 and $\bigcirc$: sample A1. Inset: the *ac* χ of powder made from sample A1 with particle size of $\bigcirc$: 1.9 μm; $\bigtriangledown$: 1.3 μm; and $\square$: 0.9 μm. The solid lines are fits and the dashed line is the estimated magnetic background.

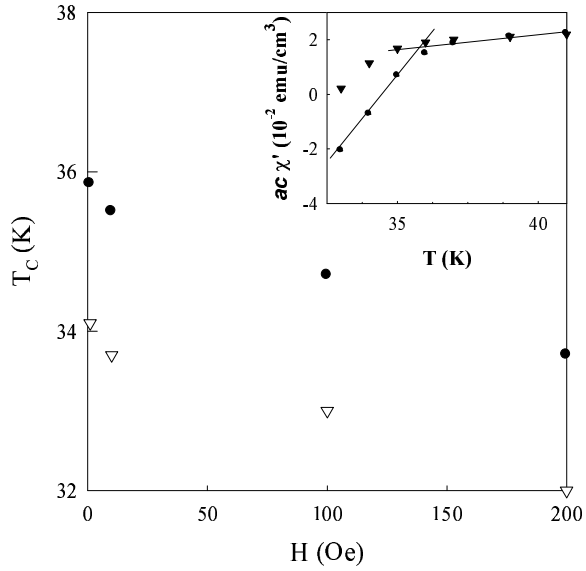

FIG. 5: The intragrain T_c, deduced as the onset of the differential χ ($\bullet$) and the inflection point of M_{FC} ($\bigtriangledown$). Inset. The differential susceptibility measured as the *ac* χ at $H_{ac} = 0.3$ Oe with a *dc* bias of 1 ($\bullet$) and 100 Oe ($\blacktriangledown$). The T_c is determined as the cross point of the linear fits (the solid lines) below and above T_c.

the comparable 300 K resistivity of the samples A0 and A1, *e.g.* ≈ 0.024 and 0.029 Ωcm respectively, suggests that m^* may not play a major role here. To verify the possibility of the simple pair-broken mechanism, the $1/\lambda^2$'s of several samples with T_c ranging from 17 K (sample C3) to 40 K (sample A1) were measured (Fig. 6).[22] In typical cuprates with pair-breakers (*e.g.* Zn), Nachumi *et. al.* have observed that T_c is a universal linear function of $1/\lambda^2$.[10] Although there are still disputes about the data-details at high Zn-levels and their interpretation, all reported T_c data approach zero with the suppression of $1/\lambda^2$ with pair-breaking. In particular, both the strong-coupling d-wave model in the unitarity limit and the "swiss cheese" model predicts $T_c << 10$ K when $1/\lambda^2 \approx 0.3$ μm^{-2} (*i.e.* $\sigma \approx$

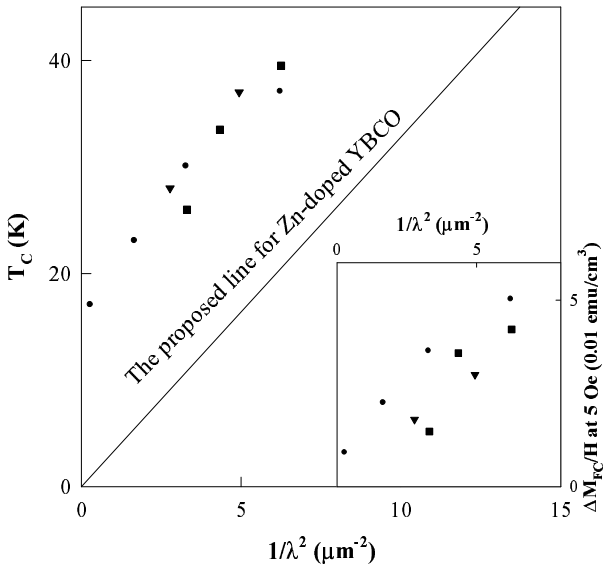

FIG. 6: T_c vs. $1/\lambda^2$ at 5 K for several annealed samples. Solid line: the Uemura line. Inset: the diamagnetic drop $\Delta M_{FC}/H$ across T_c at 5 Oe vs. $1/\lambda^2$. Symbols used are ■: A0-A2; ▼: B0-B1; ●: C0-C3.

0.02 μs^{-1} in a μSR measurement).[10,23] It is, therefore, interesting to note that all of our data points fall on the far left of this line. In particular, the T_c of sample C2 with $1/\lambda^2 \approx 0.3$ μm^{-2} is still 15 K or higher. The trend is in line with the data of Ru1212, where samples with λ(5 K) as large as 2-3 μm still have $T_c > 20$ K.[12] A simple pair-broken mechanism, therefore, may have difficulty in accommodating the data. This view is supported by the fact that there is no systematic correlation between the T_c (or $1/\lambda^2$) and the M_{FC} (or the remanent moment) above T_c, *i.e.* the FM aligned spins. In fact, the M_{FC} at 5 Oe and 50 K differs less than 5% between samples C3 and C1 with T_c of 17 and 37 K, respectively. The M_{FC} spread for samples A0, A1, and A2 is slightly larger, but again without a systematic dependency. A similar trend has also been pointed out previously in both the Ce-doped Ru1222 and the Cu-substituted Ru1212.[8,15] Intragrain granularity, therefore, seems to be a more reasonable interpretation. It was proposed that the T_c of a JJA is $2.25J$, with J being the coupling energy of a junction.[24] The λ, in such a case, may depend on the length of the junctions involved, but the T_c will not. A non-zero phase-lock temperature, therefore, may coexist with an unusually long λ if the junction length is large.

Granularity of Ru1212 has been previously reported and attributed to either structural defects or possible phase-separation.[1,2,12] However, no correlations between the T_c and the proposed 90°-rotated *a*-*c* microdomains (or the coherent rotation of the RuO_6 octahedrons) have ever been observed.[1,25] We, therefore, favor a mesoscopic phase-separation between FM and AFM species as the origin of the granularity.[11,26] The observation of a correlation between the magnetic transition temperature and the granularity (T_c, $1/\lambda^2$...) here certainly supports this view (Fig. 2). However, we will not discuss it further since it is not essential for the topics concerned, *i.e.* the evolution of T_c and Meissner effect with granularity.

The systematic increase of ΔM_{FC}, the Meissner effect, with annealing is also obvious in Fig. 2. The large Meissner fraction in a Ru1212 sample below 1 Oe and its disappear once above 10 Oe has previously been taken as evidence for a homogeneous superconductivity and a spontaneous vortex state (SVS), respectively.[1] The interpretation, in our opinion, is neither the only possible one nor the most likely one. Within the framework of the Ginzburg-Landau (G-L) model, the reversible part of ΔM_{FC} of a type II superconductor should have a maximum of $H_{c1}/4\pi$ at H_{c1}; and a few times smaller ($\approx \frac{\phi_o ln(H_{c2}/H)}{32\pi^2\lambda^2} = \frac{H_{c1} ln(H_{c2}/H)}{8\pi \ln \kappa}$) in mixed states far above H_{c1}, where H_{c1}, H_{c2}, and κ are the lower- and upper-critical fields and the G-L parameter, respectively.[27,28] This is simply the result of a competition between the magnetic energy M·H and the carrier kinetic-energy, and should hold even in the existence of spontaneous vortex and an internal magnetic field B_M ($= 4\pi M$ in homogeneous ferromagnetic superconductors). In particular, the ΔM_{FC} should be $\approx \phi_o \ln \kappa/(16\pi^2\lambda^2)$ (> 1 emu/cm^3 with $\lambda < 0.5$ μm) over a broad H-range regardless of the value of B_M if the pinning is weak. The maximum ΔM_{FC}, we would argue, is a far better parameter for ferromagnetic superconductors than the widely used χ, whose interpretation may be ambiguous due to the uncertainties in the B_M and the possible SVS. The ΔM_{FC} of sample A1, for example, is ≈ 0.3 emu/cm^3 at 20 Oe, and seems to increase continuously with H although the large magnetic background makes a quantitative estimation difficult at larger fields.

This value is not too far from that expected based on the deduced $\lambda \approx 0.4$ μm, considering the corrections needed for the random grain-orientations and vortex pinning. The much smaller $\Delta M_{FC} < 0.04$ emu/cm^3 over the whole H range reported) in Ref. 1, on the other hand, may imply a unusually long λ, *i.e.* severe granularity, if the pinning is not too strong.

It should be noted that a 100% Meissner effect can be reached in a JJA-like heterogeneous superconductor below its effective $H_{c1} \propto 1/\lambda^2$. The value of $\Delta M_{FC}/H$ is determined by both a free-energy balance and the vertex pinning. For the energy balance, all of the conclusions of Hao *et al.* should still hold if the JJA parameters are used.[27] In particular, a full Meissner effect can be expected below $\phi_o/(32\pi^2\lambda^2) \approx 1$ Oe with a λ as large as 2 μm. The pinning strength, on the other hand, depends only on these parameters averaged over a length-scale of vortex cores. The $dT_c/dH >$ 100 K/T observed in Ru1212 powders suggests that the cores of the related Josephson vertex may be as large as 10^{-2}-10^{-1} μm.[14] The pinning, therefore, can be very weak if the sample can be regarded as homogeneous over such length-scales. The fact that full Meissner effects have been routinely observed in both underdoped and overdoped cuprates, where evidence for a possible mesoscopic phase-separation is accumulating,[29] supports the arguments. To verify this, the $\Delta M_{FC}/H$ of the nine samples at H = 5 Oe is plotted against their $1/\lambda^2$ at 5 K (inset, Fig. 5). The rough linear-correlation between the two parameters indicates that the Meissner effect $\Delta M_{FC}/H$ of Ru1222 is mainly determined by the intragrain Josephson penetration depth λ, at least for the samples examined here. Notice that the largest $\Delta M_{FC}/H$ observed here (0.06 emu/cm^3 at 5 Oe) is already a significant fraction of $1/4\pi$; a 100% Meissner effect below 1 Oe may not be sufficient evidence for microscopic-homogeneous superconductivity.

In summary, the intragrain T_c of Ru1222 has been tuned from 17 K to 40 K through O_2/Ar annealing. The corresponding change in the normal-state hole-concentration, however, is too small to account for the change. The associated intragrain $1/\lambda^2$, on the other hand, increases 20-fold, much more than that expected from the proposed T_c *vs.* $1/\lambda^2$ line for homogeneous cuprates. A Josephson-Junction-array model, therefore, is invoked to interpret the data. The increases in both T_c and Meissner effect with oxygen-intake in such a model are mainly due to the improvement of the intragrain granularity.

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